

THE CONCENTRATION
OF
GOLD AND SILVER IN IRON BOTTOMS.

PRECIPITATED FROM HIGHLY FERRUGINOUS COPPER MATTE.

BY

MYRICK N. BOLLES, B. S.

DISSERTATION

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF DOCTOR
OF PHILOSOPHY IN THE FACULTY OF APPLIED SCIENCE OF
COLUMBIA UNIVERSITY.

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ACKNOWLEDGEMENT.

This investigation has been carried out under the direction of Professor Henry M. Howe, of the Department of Metallurgy, Columbia University, to whom I take great pleasure in expressing my gratitude for his ready council and suggestions. I also wish to acknowledge my indebtedness to Mr. Bradley Stoughton, Instructor in Metallurgy at Columbia University, for his interest, and to Mr. Theodore Knutzen, Superintendent of the National Smelting Co., of Rapid City, So. Dak., for furnishing the material upon which this research has been conducted.

M. N. B.

Metallurgical Laboratory, Havemeyer Hall,
Columbia University,
May, 1903.

BIOGRAPHICAL.

Myrick Nathaniel Bolles entered the State College of South Dakota in May, 1894, where he received the degree of B. S. in Mechanical Engineering June 1898. He entered Columbia University October, 1901. Studied under the Faculty of Applied Science for the degree of Ph. D. until June 1903.

The Concentration of Gold and Silver in Iron-Bottoms.*

BY MYRICK N. BOLLES, B.S., NEW YORK CITY.

(Lake Superior Meeting, September, 1904.)

INTRODUCTION.

THE concentration of gold and silver in mattes low in copper, and the subsequent separation and recovery of either or both of these metals, is a question the satisfactory solution of which has long vexed metallurgists. It covers a broad and important department of metallurgical science and brings in its train a series of attendant problems continuing from the initial operations of concentration to the final recovery of "values."

The particular phase of metallurgy which perhaps is most vitally associated with this series of problems is the so-called pyritic smelting for the reduction of gold- and silver-ores. This association follows from the conditions which govern the operation of the majority of such plants and under which they must be expected to remain while they continue to occupy their present and, what seems to be, their natural position in the scheme of metallurgical economy.

The principal object of these plants being, as a rule, the use of low-grade matte as a carrier for either gold or silver, and these two metals being the source of profit, it follows that a matte is usually made which is too rich in these metals to be allowed to follow the ordinary course of treatment to which copper-matte, for instance, is subjected. It may be, and often does happen, that such matte is produced in operations which must be classed under modes of treatment other than pyritic, but these cases are the exception and not the rule. In such cases, however, the subsequent treatment of the matte produced becomes of great importance.

* This paper was submitted in partial fulfilment of the requirements of the degree of Doctor of Philosophy in the Faculty of Applied Science, Columbia University, New York City; and at the same time was accepted by the American Institute of Mining Engineers for publication in the *Transactions* of that Society.

The most advantageous conditions for such pyritic smelting seem to be where refractory gold-bearing ores of a smelting grade occur remote from lead- or copper-deposits, or where the cost and scarcity of such ores prohibit their free use as carriers and collectors of the precious metals.

In such cases to escape the expensive resort of shipping the gold-bearing ores to a more favorably placed plant, recourse is often had to one of the varieties of pyritic smelting if the ore is rich in sulphur; or, if it is oxidized or "dry," sufficient low-grade or even barren pyrites are charged, and the operation conducted quite as in any matting operation in the blast-furnace. In either case, some copper-ore is usually charged and the result is ferruginous matte carrying more or less of the precious metals, as the case may be.

The copper-ore charged in order to produce this matte is in such cases often treated at a loss on account of the expense of shipping it to the plant,¹ hence it is usually the aim of the smelter to produce a product as low in copper as possible.

As yet no satisfactory method of direct treatment of such matte (that is, matte containing less than 10 per cent. copper, and from 20 to 40 oz. gold with perhaps 100 oz. silver per ton) seemed to have been evolved, and for this reason it is found most convenient to dilute it largely by introducing it in small quantities into some one of the more ordinary metallurgical operations which is not over-burdened with an excess of gold or silver.

The usual practice is to ship it to a lead- or copper-smelter where it is charged, either raw or roasted, with a large percentage of the regular charge, and the precious metals eventually recovered, from the resultant metallic lead by the Parkes process, or from metallic copper as vitriolization- or electrolytic-slimes.

The smelting charge for the treatment outlined above is usually high, in addition to heavy freight-rates for transportation. The copper-content causes the production of lead containing a large quantity of very objectionable copper as an impurity; and a large quantity of copper-matte carrying sufficient lead, so that the subsequent and heavy loss of this latter metal

¹ F. R. Carpenter, *Trans.*, xxv., 772.

upon retreatment is a serious item to the lead-smelter. On the other hand, it is rather low in copper, and too high in gold and silver, to be acceptable to the copper-smelter. Thus it is a difficult and expensive product to place on the market.

Local treatment as a rule is out of the question, as it is too low in copper and too high in the precious metals either to bessemerize or to concentrate by roastings and smeltings in the reverberatory furnace.

METHODS OF DIRECT TREATMENT.

Direct treatment is the "open sesame" of the situation, and as the problems of initial concentration to the ferrous matte have been fairly well mastered, the recovery of the contained precious metals remains as the residual matter of importance.

To effect the separation of gold and silver from ferrous or cuprous sulphide (leaving out the field of hydro-metallurgy entirely), recourse may be had to two distinct and radically different operations.

1. By the difference in affinity (that is,—to express more definitely, the heats of combination) of the constituent elements for an outside agent (more often oxygen assisted by silica, etc.), they can be successively removed, and this concentration carried to any extent desired, or until the recovery of the remaining metal is comparatively simple.

2. By treating the matte either solid or liquid with an insoluble molten body having a greater attraction than the matte itself for gold and silver, and thus remove them more or less completely by absorption.

Of the two courses the latter seems to offer distinct advantages for experimental research, inasmuch as the first with various modifications is in constant use at the present time at different reduction works, and its limitations are quite well defined.

To have a possibility of application, the absorbent must be obtainable at a price not too excessive. Its heat of combination with sulphur should be lower than those of iron and copper, lest by reaction it be converted into a sulphide. It must be comparatively insoluble in matte to avoid its excessive loss and possible interference with subsequent treatment of the matte. It should possess great power of absorption so that the gold or silver may be removed as completely as possible.

The following substances may be selected from the elements and their compounds as possessing to a degree qualifications such as to render them tentatively suitable as the absorbent bodies, namely, metallic sulphides, etc., aluminum, lead, copper and iron.

METALLIC SULPHIDES, ETC., AS ABSORBENTS.

The metallic sulphides, arsenides, antimonides, and perhaps a few rarer compounds of the same general nature, possess properties which render them of peculiar interest in this connection. Those of lead, iron and copper are useful carriers of the precious metals in furnace practice. Being stable compounds there is not the same tendency of reaction that there is in the case of metallic absorbents in contact with sulphides.

As it is from metallic sulphides that absorption must be made, it follows that to apply the sulphides, etc., as absorbents, there must exist a considerable difference in their attraction for gold and silver. That this exists is shown by the slight affinity between gold and pure ferrous sulphide,² whereas in the impure state of ordinary matte it is a very powerful absorbent. These differences in absorptive power exist in the antimonides and arsenides as well, and may no doubt be intensified by proper treatment.

The extreme miscibility of the sulphides, and to a certain extent also the solubility of the speisses in each other and in sulphides, is the main factor which prevents the application of these bodies for the purpose of extracting gold and silver from matte. It remains to be seen whether by proper treatment the miscibility of the sulphides, etc., cannot be overcome and thus allow their use as absorbents. The field for investigation in this direction is indeed a broad one and one which promises valuable results.

In an experimental way some half-a-dozen fusions were run upon different sulphides in an effort to obtain a stratification of the matte, but the action of diffusion was too great to allow of such a separation being accomplished with ease.

² This section is discussed more fully later on in this paper under the heading "Metallography of Mattes."

ALUMINUM AS AN ABSORBENT.

Aluminum and gold combine with great avidity to form alloys of the same class as the purple one discovered by Sir Wm. Roberts-Austen, the heat of the combination being calculated by Richards,³ from an experiment by Roberts-Austen, to be about 1,300 kg-cal. per kg. of aluminum, and on account of this avidity for gold it was hoped that aluminum might prove to be an efficient absorbent from matte. Aluminum, however, possesses a heat of combination with sulphur, to form aluminum sulphide, Al_2S_3 , which is far greater than that of either copper or iron. For iron-matte this relation would be as 23,576 g-cal. (the heat of formation of a gram-molecule of ferrous sulphide) is to 124,401 g-cal. (the heat of formation of a gram-molecule of aluminum sulphide), showing the probability of a strongly exothermic reaction between metallic aluminum and ferrous sulphide. In the face of this fact it might yet be possible to heat aluminum up to such a point that it would absorb gold, and yet not sufficiently high to induce the reaction.

Experimentally, this hope was shown to be vain for, at a low red heat, combination takes place readily, and aluminum-sheet heated in contact with matte to a point slightly lower than that needed for reaction to occur, was not found to contain an appreciable quantity of gold.

The reaction which takes place between metallic aluminum and this matte is mainly confined to the ferrous sulphide present, small quantities only of copper and silicon being reduced. The products are aluminum sulphide and metallic iron containing small amounts of copper and silicon.

The principal reaction is:— $3\text{FeS} + 2\text{Al} = 3\text{Fe} + \text{Al}_2\text{S}_3$.

This reaction takes place when the mass has been heated to the neighborhood of 700°C. , and is accompanied by vivid light.

The heat liberated is indicated roughly in the following thermal equation:—



$$* (+ 70728) + (- 124401) = (- 70728) + (+ 124401).$$

* These heats of combination are taken from Richard's *Aluminium*, p. 235.

³ Richard's *Aluminium*, p. 502 (1896).

The data given in the above equation correspond to 319.48 g-cal. per gram of iron separated.

The above reactions presuppose that aluminum is not added in excess of the ferrous sulphide present, otherwise copper also will be reduced. The iron which is reduced settles as a button to the bottom of the crucible, and the discovery of this reduction of iron from mattes proved to be of value later in the study of iron-bottoms.

LEAD AS AN ABSORBENT.

The use of lead as an absorbing agent is probably the oldest method of extracting gold and silver from matte. Processes accomplishing this end are described in all of our older textbooks upon metallurgy, but, outside of regular lead-reduction methods, few are in practice to-day on account of the waste of lead incurred, as well as the contamination of all of the products with lead, copper and sulphur.

Matte may be treated with lead for the purpose of extracting the precious metals; by the soaking process, in which the lead is molten but the matte is not (a representation of which is the Crooks process);⁴ by smelting with lead-ores, as in ordinary practice; or, as has been proposed, by the Davies or Probert process,⁵ in which both lead and matte are molten. The lead is introduced into the molten matte-bath as oxide and subsequently reduced, or it may be introduced as granulated metal. In both cases the lead and matte are thoroughly mingled by agitation and allowed to separate by settling.

In a description of Deadwood practice Collins says:⁶ "The matte is worked up by raw smelting with lead ores, as in the ordinary lead-smelting practice. In doing so it is worthy of note that it takes up silver from the charge while giving up nearly all its gold to the lead bullion."

In order to ascertain the behavior of this matte toward lead, two charges were made containing respectively 10 per cent. and 20 per cent. of lead oxide (lithage) and fused. The fusion was stirred with a wooden stick to ensure thorough mixing and

⁴ Collins, *Metallurgy of Silver*, p. 317 (1900).

⁵ Eissler, *Metallurgy of Argentiferous Lead*, p. 184 (1891).

⁶ Collins, *ibid.*, p. 268.

then cast. The results of these fusions are indicated in Table I., experiments Nos. 84 and 86. This operation may be taken as representing the application of the Probert process to the matte. The results yielded are approximately the same as those given by melting the matte upon a bath of lead in a reverberatory furnace, as was done in some experiments at the Deadwood plant.

The results of metallurgical experience indicate that it is not possible to bring lead and matte in contact in such a way as to effect an absorption of the precious metals without also absorbing copper and likewise contaminating the matte with lead to a serious extent.

This is the greatest objection to this type of process, as the percentage of absorption is satisfactory enough, so that the matte could be again charged into the matting furnace were it not for the loss of lead thereby incurred.

COPPER AS AN ABSORBENT.

The old Swansea extra process for the production of "best selected" copper is the most notable application of copper as an absorbent from matte. This process has been studied by Mr. Allan Gibb⁷ and also by Mr. Edward Keller⁸ and a large mass of exact data is at hand representing the deportment of metallic copper toward impurities, including gold and silver, in copper-mattes of high copper-tenor.

Mr. Gibb found in his research that "with a reduction of 8.2 per cent. of the total copper as bottoms, they contain 41.5 per cent. of the total gold; whilst, when 14.4 per cent. of the copper is reduced to bottoms, the whole of the gold is found in them. In some cases traces of gold remained in the regulus; but they were too small to be weighed, even when 50 grams of the samples were taken for assay." "Silver,—silver nearly reaches its maximum concentration in bottoms when 19 per cent. of the total copper is separated in this form,—42.9 per cent. of the total silver being then contained in them." . . . "It is worthy of note that its concentration is lessened by the

⁷ Best Selected Copper. Third Report of Alloys Research Committee, *Institution of Mechanical Engineers*, p. 254.

⁸ Elimination of Impurities from Copper Mattes, *Mineral Industry*, vol. ix., p. 240 (1900), and same title, *Trans.*, xxviii., 127 (1898).

presence of arsenic and nickel," as is shown when the copper in the bottom is 19.3 per cent., it contains only 26.7 per cent. of the total silver. The disturbing factors in this case are shown in the following analyses:

Analysis of copper-bottom: Cu, 71.31; Ni, 10.34; As, 10.91 per cent.

Analysis of regulus: Cu, 66.56; Ni, 2.52; As, 0.70 per cent.

It must be noted, as is illustrated above, that the influence of other elements, than those under study, and which may be present through intention or otherwise, must be constantly borne in mind and observed in order that the experiments shall be definite and to the point. A slight difference in the method of preparation or manipulation may have as great an effect also, and it is always wise to provide for this contingency by comparing the results of say two methods of preparation, etc.

Most of the mattes studied contained a percentage of copper approaching that of pure cuprous sulphide (79.82), in which copper is very insoluble. This insolubility does not hold for matte containing a large percentage of ferrous sulphide and in matte containing Fe 60 per cent., and Cu 10 per cent., a reaction occurs with metallic copper similar to that with aluminum resulting in the separation of metallic iron and the production of copper sulphide. The readiness with which this reaction takes place bars metallic copper from being used as an absorbent for the matte which we have under consideration.

IRON AS AN ABSORBENT.

The study of iron-bottoms reduced from these mattes was suggested by a variety of circumstances, chief among which were: 1. The fact that when such ferruginous mattes are produced in conjunction with the treatment of gold- and silver-ores, great masses of metallic iron, or "sows" assaying high in gold, often collect in the blast-furnace bottom. 2. The parallel concentration which is effected by copper-bottoms thrown down, as exemplified in the Welsh process for "best selected" copper. This separation in copper-bottoms has been ably studied by Mr. Allan Gibb⁹ and also by Edward Keller,¹⁰ and the behavior of gold and silver toward copper-bottoms is fairly well known.

⁹ *Loc. cit.*

¹⁰ *Loc. cit.*

On account of their capacity for absorbing gold, it is very desirable to learn more in regard to the behavior of the iron sows which collect in the bottom and forehearth of the matting furnace.

Dr. F. R. Carpenter¹¹ says of them: "They were virtually blocks of solid metallic iron weighing from 20 to 40 tons, and carrying sometimes 20 oz. of gold per ton." This is in accordance with my experience while in the laboratory of the Deadwood plant, excepting that as I remember the sows usually assayed somewhat higher.

In regard to iron-bottoms it may be of interest to quote Dr. F. R. Carpenter in regard to the practice while he was superintendent of the Deadwood & Delaware Smelter, now the Golden Reward, Consolidated Gold Mining & Milling Co. of Deadwood, So. Dak. He says: "It was my theory that the metallic iron helped to clean the slags, and I did not really care to overcome the formation, but others have tried to do so. It was my theory that if the gold was not recovered in the sows, it would be lost in the slags, and here is, in my opinion, the solution of the question of clean slags in the use of iron matte free from copper."

Little or no data of an exact or numerical nature seems to have been published upon the function, the relation, and possibilities of this metallic iron in mattes of this character, and it was the original object of this investigation to ascertain as nearly as possible the availability and rate of concentration of gold and silver in iron-bottoms thrown out of a commercial matte of this type.

This portion of the research has been carried out in a manner very nearly parallel to the laboratory experiments of Mr. Allan Gibb described in the article formerly referred to. This parallelism has been maintained on account of its wide application to cases of absorption phenomena, and because its clear, direct application renders its deductions easily grasped by a comparatively non-technical mind.

The above renders the results of each research strictly comparable with the other, and it is to be hoped that any future investigations may be capable of a like presentation.

¹¹ *Trans.*, vol. xxx., 764 (1900).

SCHEME OF PROCEDURE.

Of the five substances tentatively assumed as suitable for use as absorbents, iron would seem to be the most inviting. It is low priced and available in nearly all localities; it possesses a heat of combination with sulphur only slightly greater than that of copper; it is not extremely soluble in ferrous matte, and it possesses a superior affinity for gold. All of which are necessary for its successful application as an absorbing medium, and for purposes of research it presents one very necessary attraction, that is, a lack of data as to its availability and behavior.

With a view of investigating this phase of the question, it was decided to separate the iron contained in a standard matte in various percentages, varying from 10 to 75 per cent. of the total iron. This iron would be collected in a button or so-called "bottom," from the assay of which, together with that of the regulus, the distribution of the gold in the two portions could be readily ascertained.

In order to guard against the chance of being seriously misled by the influence of the precipitating reagents, at least two quite unlike methods of reducing the bottom should be employed.

Actually five methods were used, namely: I. Poling at a high temperature. II. Reduction by metallic aluminum. III. Reduction by potassium ferrocyanide. IV. Reduction by metallic copper. V. Addition of metallic iron.

The matte upon which these experiments were conducted was furnished by the National Smelting Co. of Rapid City, So. Dak. It is as nearly a duplicate of the matte produced by the well-known Deadwood and Delaware Smelter, now the Golden Reward Consolidated Gold Mining and Milling Co. of Deadwood, as products from different works can be. It is produced under the same conditions from ores of the same district, by the same type of plant, and by men who gained their experience in the original Deadwood works. The object is the same at both works, namely, the recovery of gold and silver from the refractory siliceous ores of the Bald Mountain, Ragged Top and Ruby Basin districts.

These ores contain 80 per cent. or more of silica, and are smelted with a flux of dolomitic limestone, and the addition of sufficient barren pyrite to form a matte of the required tenor.

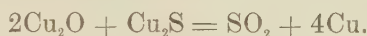
The slag is remarkably high in silica. An approximate analysis of the Deadwood slag given from memory is: SiO_2 , 50; FeO , 11; Al_2O_3 , 6; CaO , 23; MgO , 10 per cent.

Due to the silica-content and the consequent elevation of the fusing-point a very high temperature is necessary in the lower part of the furnace, and a powerful reducing action results. To these conditions is probably due the character of the matte and its remarkable cleaning action on the slag.

Following is the analysis of the matte experimented upon.

Fe, 61.68; Cu, 11.205; S, 25.47; SiO_2 , 0.662; Au, 0.00457; Ag, 0.0092; Pb, 0.050; Zn, 0.00; Ba, 0.047; As, 0.22; Sb, 0.00; Mn, 0.00; Sn, 0.00; Ca, 0.31; Total, 99.66577 per cent.

I. *Reduction by Heating or Poling.*—Copper bottoms are obtained from rich, partly roasted, copper matte by simple fusion, resulting in the precipitating of metallic copper, according to the following reaction:—



This result cannot be obtained in the case of a ferrous matte, as was demonstrated by the fusion of varying mixtures of raw and roasted matte.

Charcoal powder was tried, and also poling with a green stick, to simulate the reducing action of the furnace. In all of these fusions, which were brought to about $1,400^\circ \text{C}$,¹² molten matte occupied the lower portion of the crucible, while above floated iron oxide partially slagged by its attack upon the silica of the crucible. The cast of this melt yielded only matte and slag.

Thus the attempt to reduce metallic iron from slightly oxidized matte by poling was not successful, but it was found that a simple fusion of the raw matte at a high temperature yields a nearly constant percentage of bottom, being about 6.5 per cent.

Bottoms designated in Table I. as No. 11 and No. 25 were obtained in this manner.

II. *Reduction by Metallic Aluminum.*—The reduction of iron was attempted by the use of powdered aluminum so as to secure a very intimate contact of the reduced iron with the mass of

¹² On account of the extremely corrosive action of these iron-mattes upon the protecting tube of the thermo-couple, the exact temperature was not measured, but a close approximation was secured by allowing the thermo-junction and tube to rest just above the surface of the matte for a few moments. Even under these precautions the spray constantly thrown off proved to be very destructive.

the matte, but the use of the powdered form was found to be objectionable, and metallic aluminum rods were substituted with success.

The aluminum sulphide produced is difficultly fusible, and if the quantity of aluminum added in the powdered state is sufficient to cast down an iron-bottom, the mass in the crucible is converted into a refractory clinker.

If less than about 5 per cent. of metallic aluminum be added, no iron is cast down, but above this point the bottoms rapidly increase in size until the equivalent of iron is reduced.

The fusion was conducted as follows: The weighed portion of matte was melted in a graphite crucible, and into this the rod of aluminum was plunged, and slowly fed downward with a stirring motion, as it was consumed.

No difficulty was experienced with the fusion, and it remained perfectly fluid after the introduction of as high as 15 per cent. of aluminum. The crucible was allowed to stand for 5 min. and poured into a large conical iron-mold. After cooling, the whole was weighed and the iron-bottom broken out, carefully cleaned from the adherent matte, weighed and crushed in a diamond mortar for sampling.

The residue, composed of sulphide of copper, aluminum, iron, etc., very rapidly disintegrated in the air with the evolution of hydrogen sulphide (H_2S), due to the reaction of aluminum sulphide upon atmospheric moisture, as follows:—



To avoid change of weight the samples were assayed immediately.

Aluminum sulphide does not readily mix with the other sulphides of the matte, and is often found at the top of the cast as a distinct, lighter layer, assaying low in gold and silver.

III. *Reduction by Means of Potassium Ferrocyanide.*—As the result of a number of experiments, it was found that if a charge of finely ground matte be mixed with ground potassium ferrocyanide and fused, the following changes take place progressively:

1. Gradual decomposition of the ferrocyanide takes place with the evolution of ammonia fumes, due to the reaction of

the nitrogen of the ferrocyanide with its water of crystallization.

2. A process of liquation takes place, resulting in a porous crust floating on molten sulphides.

3. On further heating or stirring, this crust dissolves in the sulphide, and on pouring, a bottom or metallic portion is rarely found.

4. If this crust be separated in the second stage by pouring and skimming back the crust, and then melt it down alone, it will result in the major portion separating as a metallic bottom covered with a thin layer of sulphide.

In this manner were prepared the bottoms in Group II.

It is possible thus to prepare almost any desired percentage of bottom, but the percentage will depend not only upon the quantity of ferrocyanide used, but also upon the temperature employed, and the time of heating after liquation, and before skimming. Long heating and high temperature tend to produce small bottoms.

The mattes after treatment are greatly changed. The fracture becomes columnar and highly crystalline, with broad cleavage surfaces. This change is due to the introduction of the alkali metals, in this case potassium, doubtless as the sulphide. This is demonstrated by melting matte with potassium sulphide, when perfect intermixture takes place with the above noted change in fracture. It is worthy of note that pure copper sulphide shows the same deportment with potassium sulphide.

A very rapid oxidation of these mattes takes place upon exposure to air, with consequent disintegration. This oxidation is so rapid that freshly ground matte will rapidly attain a temperature of 60° C. if exposed to air. It is greatly hastened with proportionate increase in temperature by directing a gentle stream of oxygen upon the matte.

In experiment No. 81, lead oxide (PbO), equal to 10 per cent. of the matte-charge, was added in addition to the ferrocyanide, in order to observe the influence of lead-bearing mattes upon the absorption of gold and silver in the iron-bottom. The fusion was conducted without skimming and an iron-bottom was found in the cast, free from any accompanying reduced lead. It will be seen upon referring to Table I. that this addi-

tion of lead oxide which combined to form matte is practically without influence upon the absorption.

The original matte undergoes a liquation with the separation of a crust similar to that just described, but the crust in this case does not yield a metallic bottom. Three of these crusts were separated by skimming, and as they give results almost identical they are represented by a single sample, designated as No. 61 in Table I.

IV. *Reduction by Metallic Copper*.—The addition of copper was made with the intention of obtaining copper-bottoms. The charge for experiments Nos. 68 and 70 was made up of finely ground matte, to which was added 30 per cent. of the weight of fine shot-copper and a liberal excess of charcoal-dust to prevent oxidation. The casts from these fusions revealed upon examination not a copper-bottom, but an iron-bottom which clung tenaciously to the matte.

The second charge, experiment No. 90, was made up in the same proportions as Nos. 68 and 70, but omitting the charcoal-dust. The casts resulting did not show the separation of either metal as a bottom. However, the fracture of the regulus from these fusions showed small crystals of iron in great abundance. (Fig. 6 is a microphotograph of a polished section of No. 69.)

V. *Reduction by Metallic Iron*.—Fine iron-drillings mixed with the ground matte and fused yield a bottom of iron.

The incomplete contact with the matte, as the iron sinks during fusion, would lead one to expect the low percentage of extraction which is indicated by the line representing the results with those irons on Figs. 1 and 2, Group III.

EXTRACTION OF GOLD AND SILVER FROM IRON BY MEANS OF LEAD.

Metallic iron weighing 1,531 g., assaying 17.21 oz. gold and 5.2 oz. silver per ton, was melted in a graphite crucible, and 794 g. of test-lead was added in portions and thoroughly stirred. The resulting lead-bottom weighed 466 g. and the residual iron weighed 1,802 g., indicating that it had taken up about 271 g. of lead. The assays showed that the gold and silver had been largely absorbed by the lead, and the results designated as experiment No. 96 show the percentage of extraction. It will be seen that the transfer of gold and silver to the lead is fairly complete, but the retention of so much lead by the

iron would be a serious factor against its treatment by this method.

In obtaining the following results great care has been exercised to use accurate methods of estimation.

For the assay of the iron-bottoms the combination method was used. The sample was dissolved in dilute sulphuric acid, lead acetate and a slight excess of salt solution was added and the precipitate and residue filtered off. The dried filter paper and contents were then scorified and cupelled as usual. This method checked against the direct scorification with test-lead and litharge, and was preferred for the reason that a larger sample could be taken.

For the assay of the regulus and mattes, the crucible method was employed, using a heavy charge of litharge as recommended W. G. Perkins.¹³

The results have been classified in groups according to the method of separating the iron-bottoms. The percentages of bottom has been calculated 1. Directly as percentages of the total weight of the fusion; that is, the weight of bottom plus the weight of regulus; as is shown by the following:

$$\frac{\text{Weight of Bottom} \times 100}{\text{Weight of Regulus} + \text{Weight of Bottom}} = \text{percentage.}$$

2. As percentages of iron in bottom to total iron in regulus and bottom.

This is more logical, as it thus makes the percentage of absorption a direct function of the percentage of iron thrown down. This method was followed by Allan Gibb¹⁴ in his study of copper-bottoms, and his results can be compared directly with those herewith given.

The method of computation is shown by the following form:

$$\frac{\text{Weight of Bottom} \times \text{per cent. of Fe in Bottom} \times 100}{\text{Weight of Regulus} \times \text{per cent. of Fe in Regulus} + \text{Weight of Bottom}} = \text{percentage, x per cent. Fe. in Bottom.}$$

The results are plotted in Cartesian co-ordinates on Figs. 1 and 2. The percentages of gold and silver respectively con-

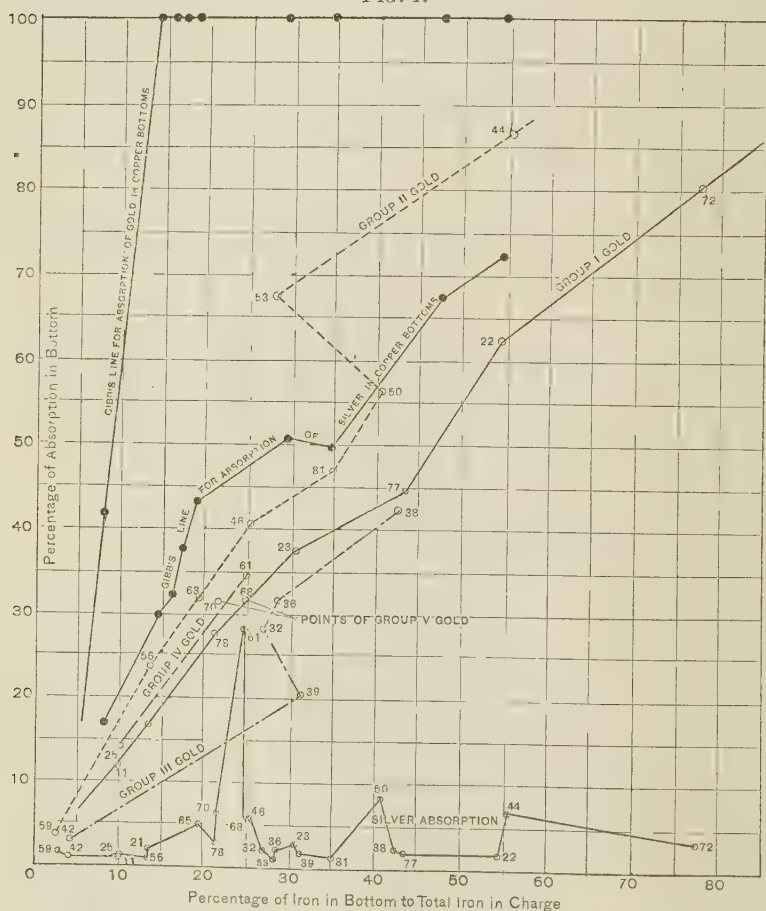
¹³ The Litharge Process for Assaying Copper Products, *Trans.*, xxxi., 913.

¹⁴ "Best Selected Copper." Fourth Report of the Alloys Research Committee, *Institution of Mechanical Engineers*, 1895.

tained in the bottom being placed as abscissa, and the percentage of bottom as ordinates.

These points have been connected by straight lines in order to facilitate inspection, and to render at once evident to the eye, the relations which the different groups bear to each other.

FIG. 1.



The grouping of the points representing silver absorption was not attempted on account of the low and variable values obtained.

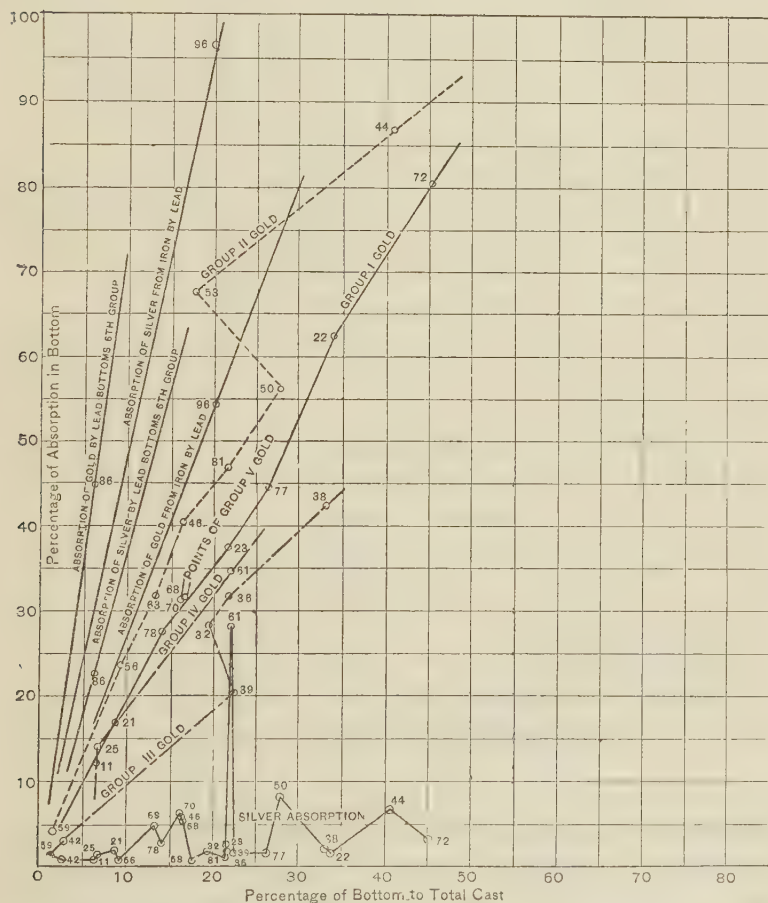
The experiment number is attached to each point plotted to render reference to the figures and tables easily made.

The lines representing absorption by lead are drawn in their probable position, but this does not constitute an attempt to

fix them in this position, as enough determinations were not made to render this point rigidly exact.

The curves obtained for silver and gold, by Mr. Allan Gibb in the research previously referred to, have been introduced for comparison on Fig. 1, in which the values obtained for gold or silver in iron have been similarly derived.

FIG. 2.



On comparing the results obtained by the different methods of precipitating iron-bottoms (Table I.), it will be seen that there is not as great a divergence as might have been expected considering the variety of reagents employed. It is seen in Fig. 1 that the points fall roughly into three sets. 1. Showing the greatest absorption, the iron-bottoms thrown down by

TABLE I.—*Results Obtained by Various Methods of Precipitation.*

Experiment No.	Weight of Regulus.	Weight of Bottom.	Quantity Cost as Bottom.	Iron.		Total Iron in Bottom.	Regulus Contains.		Bottom Contains.		Total Gold Separated in Bottom.	Total Silver Separated in Bottom.
				In Regulus.	In Bottom.		Gold.	Silver.	Gold.	Silver.		
GROUP I. Bottoms separated by metallic aluminum.												
	Grams.	Grams.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Mg.	Mg.	Mg.	Mg.	Per Cent.	Per Cent.
21	746	72	8.8	59.1	93.6	13.3	336	810	68.5	16.0	16.9	1.9
22	637	324	33.7	41.2	95.7	54.2	174	853	285.0	12.1	62.3	1.4
23	710	197	21.7	59.3	94.1	30.6	277	666	166.0	22.2	37.4	2.5
72	582	475	45.0	23.7	96.1	77.0	88	877	358.0	27.7	80.3	3.0
77	718	254	26.2	44.6	94.7	43.2	253	882	204.5	13.6	44.6	1.5
78	884	145	14.1	58.6	95.3	21.1	335	830	126.8	23.4	27.5	2.7
GROUP II. Bottoms separated by potassium ferrocyanide.												
44	628	430	40.6	52.4	93.8	55.2	68.5	791	446.0	55.8	86.8	6.6
46	750	147	16.4	53.6	91.6	25.1	258.3	787	174.5	48.5	40.4	5.8
50	810	312	27.8	53.7	94.3	40.4	221.7	853	285.6	74.0	56.1	8.0
53	819	176	17.7	51.4	92.1	28.0	153.0	895	282.0	7.8	67.2	0.7
63	810	124	13.3	58.2	90.8	19.3	308.0	876	144.0	45.2	31.9	4.9
56	895	92	9.3	61.4	91.2	13.3	344.0	880	105.0	7.0	23.4	0.79
59	947	16	1.66	60.8	89.1	2.4	420.0	885	15.5	12.0	3.6	1.3
81 (a)	819	226	21.6	45.6	88.0	34.8	237.5	922	206.5	9.3	46.8	1.0
GROUP III. Bottoms separated by addition of metallic iron.												
32	930	226	19.5	63.0	95.2	26.8	319	900	124.5	16.5	28.1	1.8
36	927	259	21.8	66.0	93.0	28.3	297	834	137.0	16.2	31.6	1.9
38	830	407	33.0	63.2	94.3	42.2	259	807	188.5	16.8	42.2	2.0
39	850	247	22.5	63.1	97.1	31.0	351	892	90.0	13.6	20.2	1.5
42	854	24	2.74	65.3	96.8	4.0	449	888	14.2	5.9	3.07	0.7
GROUP IV. Bottoms separated by high temperature or poling or liquation.												
11	607	44	6.5	62.0	92.7	9.8	244	554	33.6	4.5	12.1	0.8
25	1200	86	6.7	62.2	96.5	10.0	482	940	79.3	10.3	14.1	1.1
61 (b)	736	207	22.0	62.0	72.8	24.8	298	646	155.4	251.0	34.4	28.0
GROUP V. Bottoms separated by addition of metallic copper.												
68	1068	212	16.6	52.2	86.2	24.7	303	864	140	47.5	31.6	5.2
70	1062	205	16.2	53.1	89.5	21.5	310	842	140	54.7	31.2	6.1
GROUP VI. Lead bottoms thrown out of matte.												
84	1072	11.47	1.06				410	883	23.24	29.26	5.36	3.2
86	1053	67.85	6.05	53.9			248	698	201.96	201.3	44.9	22.4
	F	Pb										
96	grams. 1802	grams. 466	20.1				409	12.4	483.37	257.7	54.2	95.4

(a) In the presence of lead-matte.

(b) Group VII. Liquation of matte into crust or regulus.

means of potassium ferrocyanide. 2. Showing the least absorption, namely, those bottoms precipitated from mattes by addition of metallic iron. 3. The indefinite, intermediate one comprising the bottoms thrown out by metallic copper, the bottoms thrown out by high temperature, liquation or poling, and those thrown out by metallic aluminum.

From the position of the line representing the aluminum-iron bottoms it appears that the presence of metallic aluminum does not facilitate the collection of the precious metals as might have been expected from the energy of combination which

aluminum and gold exhibit. This may in part be due to the very complete reaction which occurs between aluminum and the sulphides present.

Most marked of all perhaps is the contrast (shown in Figs. 1 and 2) between the absorption percentages of gold and silver.

It is seen that while gold is carried down in a moderate degree by iron, silver is only slightly so; indeed it is very likely that the major portion of the silver carried down was not as an alloy with iron, but included with globules of matte in the bottom being an emulsion, as it were, of matte in metallic iron.

This also is sufficient to explain the irregularity exhibited in the values for silver absorption. The relation of gold and silver in the bottoms agrees very well with the results observed in furnace-practice where iron "sows" are produced.

Experiment No. 61 is an exception to this rule, as it is the crust skimmed off from a fusion of raw matte and charcoal. It is mainly composed of the crystals of iron, shown in the metallographic section, from which the sulphides have been liquated.

The percentage of iron (Table I., Group III., IV. and V.) has been changed during the process of liquation, so that after the operation the crusts contain 72.8 per cent. of iron and the liquated portion only 62 per cent.

The accompanying change in the distribution of the gold was such that 34.4 per cent. of the gold is concentrated in 22 per cent. of crust.

Referring to Fig. 2 it will be seen that the absorption of gold in iron-bottoms corresponds quite regularly (but is somewhat lower) to the absorption of silver in copper-bottoms as found by Mr. Allan Gibb.

Metallic iron is easily soluble in the mattes worked with, as is shown in the microscopic examination given later in this paper under the heading "Metallography of Mattes." The solubility increases with the temperature, and at these temperatures employed for the reduction of bottoms it was very marked. The attack of matte upon the bottoms produced a rough, dirty surface, which was very difficult to clean properly.

This re-solution would if long continued be very likely to alter the percentage of gold contained in the bottom. It being analogous to scorification, with consequent concentration. It

is likely that to this action can be ascribed the irregularity of the points plotted in the absorption values.

This view is upheld by the regularity of the results obtained in the Group I.; those bottoms precipitated by metallic aluminum, as these buttons were notably smooth and unattacked.

Conclusions.—1. Gold is collected in iron-bottoms in a ratio somewhat less than the collection of silver by copper-bottoms.

2. Not more than 5 per cent. of the silver can be expected to enter the iron-bottoms.

3. The precious metals tend to collect in the crust produced in liquation of the matte.

4. Provided other circumstances allow the use of metallic iron as a carrier, gold may be expected to be satisfactorily separated from the other products of fusion, but silver will not be satisfactorily collected.

5. Copper-bottoms cannot be obtained from mattes of this character on account of the reaction of copper with ferrous sulphide. Experiments Nos. 68, 70 and 90, point to this conclusion.

6. Aluminum does not markedly influence the absorptive power of iron-bottoms. Shown by results of first group of bottoms.

METALLOGRAPHY OF MATTES.

The chemical composition of mattes is subject to very great variations, and hence is not capable of a general quantitative expression, excepting through such wide limits that it becomes next to meaningless.

Edward Keller¹⁵ groups the various elements entering into its composition about sulphur as a nucleus, and regards the quantity of this element as nearly a constant placing the limits as 22 and 24 per cent.

Herbert Lang¹⁶ goes even further and classifies the arsenides and antimonides, known as speisses, along with the mattes under one family and designates them as sulphide-mattes, arsenide-mattes, and antimonide-mattes.

The excessive variation of which matte is capable will be illustrated in Table V. taken from Herbert Lang.¹⁷ These data illustrate the extreme variation in percentage of the ele-

¹⁵ *Mineral Industry*, vol. ix., p. 242 (1900).

¹⁶ *Matte Smelting* (1896).

¹⁷ *Ibid.*

ments named, as shown by the analysis of a number of widely varying types of mattes.

TABLE V.—*Variation in Composition of Mattes.*

Component.	Highest.	Lowest.	Component.	Highest.	Lowest.	Component.	Highest.	Lowest.
	Per Ct.	Per Ct.		Per Ct.	Per Ct.		Per Ct.	Per Ct.
Iron.....	70.47	0.136	Manganese....	3.00	0.000	Calcium ...	7.00	0.000
Copper.....	80.00	0.000	Silver.....	5.00	0.000	Barium.....	22.00	0.000
Lead.....	73.00	0.000	Gold	0.11	0.000	Sulphur....	44.00	trace
Zinc.....	11.50	0.000	Platinum.....	0.008	0.000	Arsenic.....	52.00	0.000
Nickel..	55.00	0.000	Bismuth.....	1.26	0.000	Antimony.	60.00	0.000
Cobalt..	54.00	0.000	Molybdenum.	2.31	0.000			

The profoundly complex nature of this metallurgical product is rendered evident by the number of elements shown in Table V. which may enter into its composition.

The constitution, meaning by this term, the compounds actually present and their several physical states, is even more complex.

Keller¹⁸ has shown that iron may exist as the magnetic oxide, and Munster¹⁹ and Farboki²⁰ advance the idea that metallic iron exists in solution and separates out on cooling.

This existence of metallic iron and its magnetic oxide, as well as various other compounds which have been isolated, would seem to point to a composite structure in matte. That this point is not altogether clear is rendered evident by the following question, in regard to constitution of mattes, taken from Peters,²¹ "Is it a chemical combination, a mixture or a partial alloy?" Upon this same subject Edward Keller²² says, "The true chemical character of copper-mattes does not yet seem to have been definitely determined, *i.e.*, it is still more or less of an open question whether they are true chemical compounds or mixtures such as igneous rocks."

With the idea of determining if possible some of the conditions existing in the internal structure of matte, a number of specimens were prepared and examined by the methods which have been so fruitful in the field of metallography.

¹⁸ *Engineering and Mining Journal*, November 16 (1895).

¹⁹ *Berg- und Huetttenmaennische Zeitung*, p. 195 (1877).

²⁰ *Idem.*, p. 184, 1894.

²¹ *Modern Copper Smelting*, p. 230 (1901).

²² *Mineral Industry*, vol. ix., p. 240 (1900).

A number of specimens also were specially prepared from the mattes made during the investigation of the absorption by iron-bottoms.

The apparatus employed both for the microscopic and photographic work is well described by W. Campbell,²³ to whom I wish to take this opportunity to express my thanks for his skillful preparation of several of the photographs given herewith.

Fig. 3 shows an unetched section of the original matte just as it was received from the works. In this photograph the white areas represent the iron, the dark areas are cavities and the ground-mass ferrous sulphide. Throughout this ground-mass of ferrous sulphide are distributed numerous slightly lighter colored bodies of rounded contour and smooth solid appearance. These are areas of copper sulphide (Cu_2S).

The effect under the microscope is that of a wavy striated bronze-colored ground-mass in which are set the bright white crystals of iron and the azure-blue areas of the copper sulphide.

This matte was chilled on water-cooled pipes at the works, and therefore the crystallization is not as complete as it would have been in a more slowly cooled specimen.

The general deportment and appearance of the constituents is sufficient to identify them, but as an additional safeguard the following tests were applied. 1. Nitric acid (2 per cent.) rapidly attacked the crystals of iron and blackened them. The ferrous sulphide was but slowly etched and the copper sulphide not at all. 2. A dilute solution of copper sulphate was applied, and the iron was immediately covered with a coating of deposited copper, the ferrous sulphide being slowly coated by copper. 3. Sulphuric acid (1:6) etched both the iron and the ferrous sulphide, but the copper sulphide remained unattacked.

Figures 4 and 5 show similarly prepared sections of mattes obtained during the operation of obtaining iron-bottoms, Fig. 4 being the matte from which bottom No. 59 was obtained by the use of potassium ferrocyanide; and Fig. 5 represents the matte remaining after vigorously poling the original matte at a temperature of about $1,400^\circ \text{C}$. to obtain bottom No. 25.

The forms of the crystals of iron in these two mattes are very different and indicate a difference in the system of crystalliza-

²³ Upon the Structure of Metals and Binary Alloys, *Journal of the Franklin Institute*, July, September, 1902.

tion. This may be due to the difference in temperature to which the two mattes were heated. In obtaining bottom No. 59, the temperature did not rise above $1,150^{\circ}\text{C}$. It is, however, to be noted that the cubic and hexagonal sections of the crystals have been observed in the same section of matte.

The copper sulphide in Fig. 5 was so dark a blue that it is now shown as a darker constituent in the photograph. It must not be confused with the black cavities which are present.

(The wavy lines in Fig. 5 are due to the Newton's-rings effect caused by a film of oil very difficult to remove.)

An increase of the copper sulphide constituent is seen in Fig. 6. This matte is that from which bottom No. 68 was obtained by addition of metallic copper. The copper sulphide now assumes the role of ground-mass and the ferrous sulphide is shown as dark areas.

In Fig. 7 is shown the photograph of a pure copper sulphide etched with strong nitric acid until its crystal cleavage shows in relief. The round black spots are cavities or the blowholes of steel parlance. In Fig. 8 is shown a like photograph, except that the pure sulphide has been fused with an excess of sulphur at a low heat. The analysis of the sulphide now is as follows: Cu, 78.86; S, 20.86; Si_2O and graphite, 0.2; Total 99.92 per cent. The theoretical composition is: Cu, 79.82; S, 20.18; Total 100 per cent.

This matte upon cooling was found to be surprisingly brittle so that large pieces could be crushed by the fingers. Its fracture was unusual, being along the surfaces of large elongated grains having polished black surfaces like those of gunpowder kernels. Evidently there is an excess of sulphur to be accounted for.

A metallographic inspection shows the excess of sulphur, probably in the form of CuS , lying between the crystal boundaries, and thus producing planes of weakness. One of these boundary lines is shown crossing the center of the photograph and it shows how complete is the isolation of each crystal of cuprous sulphide.

The very great insolubility of metallic copper in cuprous sulphide was shown by the analysis of the sulphide portion (upper) of a matte of pure Cu_2S fused in the presence of metallic copper, which was Cu, 79.71; S, 20.1 per cent., which approximates the theoretical percentages within ordinary experimental error.

Figure 9 shows the line of junction between metallic copper and the sulphide, and also the gobules of sulphide caught at the moment of solidification as they were rising through the metallic copper to the sulphide above.

METALLIC IRON IN MATTE.

The general impression gained from most writers upon this subject is that the iron occurring in mattes is present as included particles, held in suspension by the viscosity of the medium. That this is not true is seen from the delicate crystallization forms that the iron exhibits in the foregoing photographs. The iron must be in solution in some manner in the molten matte and separate upon cooling from this solution; perhaps by the decomposition of a lower sulphide.

Whatever may be its state in the molten matte, its presence is most important wherever iron-matte is to be used as a collector of the precious metals.

To render mattes efficient carriers it is the custom of smelters to provide for a certain percentage of copper, even though the source of the copper be such as to make its treatment uneconomical.

Often circumstances are such as to render it imperative that the copper-content be kept as low as possible, and many attempts have been made to replace it entirely by iron sulphide.

This more or less complete replacement of copper by iron has been found successful²⁴ up to a certain limit, which in practice may be taken as nearly a 5 per cent. copper-matte. The limit, however, is dependent upon the acidity of the slag produced, being lower in proportion as the slag is acid. But much below this limit varying results are found; some reports being good, and some otherwise.²⁵

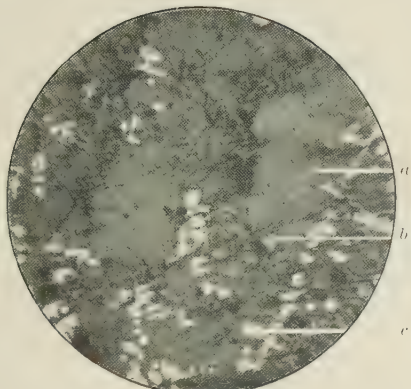
H. F. Collins²⁶ says, "The general consensus of metallurgical opinion goes to show that a pure iron-matte, FeS, is an extremely poor medium for collecting gold or silver, especially the former, though an iron-matte containing comparatively small quantities only of Cu, Bi, Fe, and As may be very efficient when the accompanying slags are of bi- or sesqui-silicate

²⁴ F. R. Carpenter, *Mineral Industry*, vol. ix., p. 696 (1900).

²⁵ Austen, *Trans.*, xvi., 262-268.

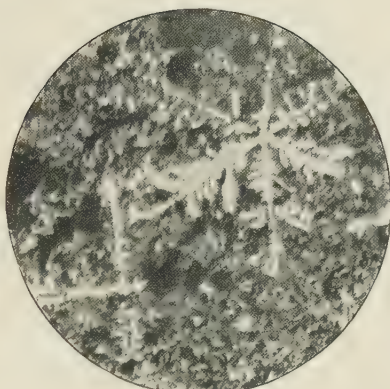
²⁶ Pearce, *ibid.*, xviii., 454 *et seq.*

FIG. 3.



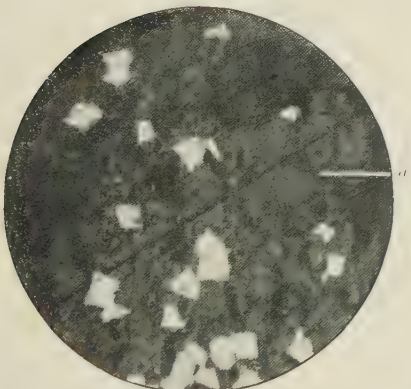
a. Iron sulphide. *b.* Copper sulphide.
c. Metallic iron.
ORIGINAL MATTE.
Magnified 130 Diameters.

FIG. 4.



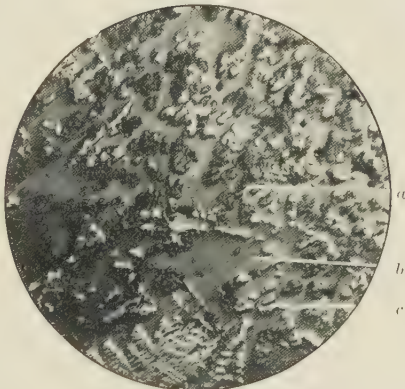
No. 59.
TREATED MATTE.
Magnified 60 Diameters.

FIG. 5.



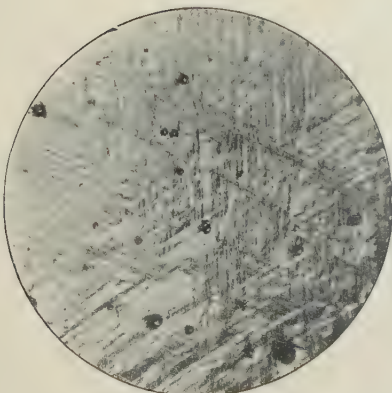
No. 25.
a. Copper sulphide.
TREATED MATTE.
Magnified 120 Diameters.

FIG. 6.



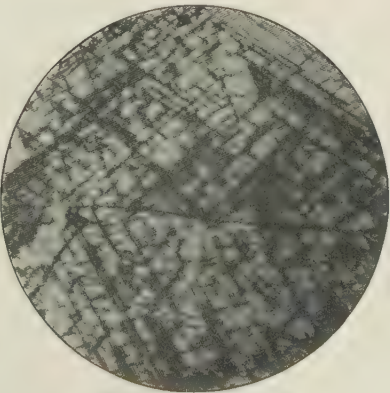
No. 68.
a. Ferrous sulphide. *b.* Copper sulphide.
c. Metallic iron.
TREATED MATTE (41.2 per cent. copper).
Magnified 40 Diameters.

FIG. 7.



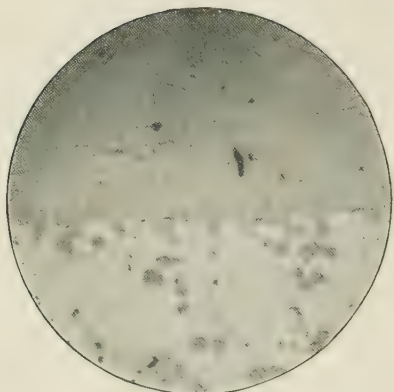
COPPER SULPHIDE.
Magnified 155 Diameters.

FIG. 8.



COPPER SULPHIDE.
Magnified 175 Diameters.

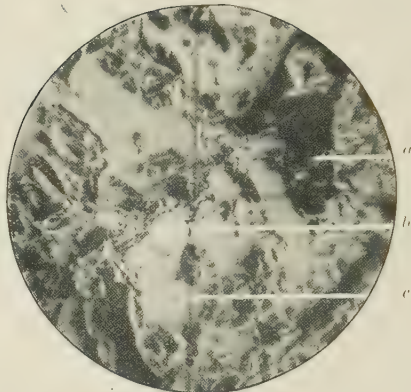
FIG. 9.



a. Matte. *b.* Copper.

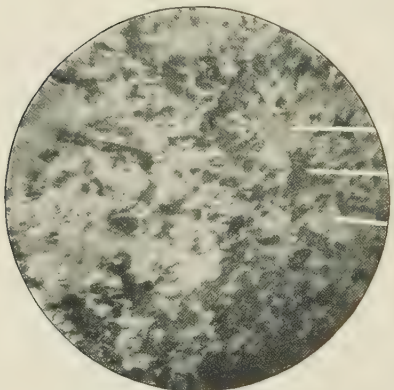
SEPARATION OF MATTE AND COPPER.
Magnified 130 Diameters.

FIG. 10.



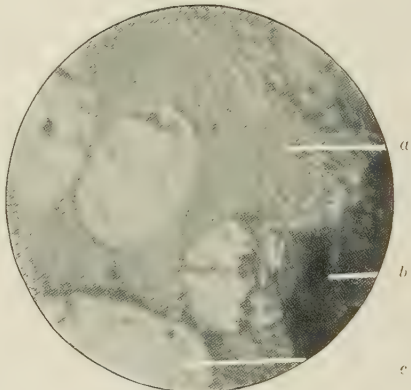
a. Cavity. *b.* Gold. *c.* Sulphide.
SEPARATION OF GOLD.
Magnified 120 Diameters.

FIG. 11.



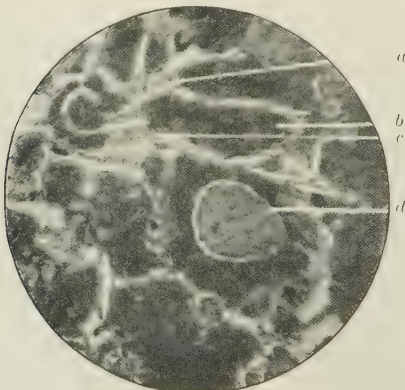
a. Eutectic. *b.* Iron. *c.* Sulphide.
EXCESS OF IRON.
Magnified 270 Diameters.

FIG. 12.



a. Eutectic. *b.* Iron. *c.* Sulphide.
EUTECTIC.
Magnified 780 Diameters.

FIG. 13.



No. 39.
IRON BOTTOM.
Magnified 130 Diameters.

type.²⁷ With slags of mono-silicate type, and with those still more basic, it seems impossible to successfully concentrate the precious metals without a considerable proportion of copper or lead in the matte."²⁸

The high silica and lime-content of the slags advocated by Carpenter,²⁹ Lang,³⁰ and Collins³¹ tend to a high temperature within the furnace, and this temperature is shown by Le Châtelier and M. Ziegler³² to facilitate the production of metallic iron in ferrous sulphide.

May not this be the point about which centers the solution of the conflict of reports as to the efficiency of iron-matte as a collector of the precious metals?

Spilsbury's³³ experiments upon reverberatory matting, and the experiments of Pearce³⁴ show the inefficiency of pure ferrous sulphide as a carrier for gold, but it is the record of Dr. Carpenter³⁵ to have obtained a very efficient action from iron-matte.

To study the action of metallic iron in mattes the following experiment was performed: An alloy of gold and pure iron was prepared by melting fine gold and pure soft iron-wire in such proportions as to yield an alloy containing 15 per cent. of gold. The micrographic section of this alloy showed it to be mainly a solid solution of gold in iron, with only small areas of a rich gold alloy showing between the grains of ferrite.

This alloy was next treated at a red heat with sulphur and converted thereby into ferrous sulphide. The bronze-colored sulphide was non-magnetic, and showed a button of separated gold on its lower surface. Fig. 10 shows a section of this sulphide and the globules of gold which are distributed throughout it. Ferrous sulphide is very difficult to polish, being cracked and filled with numerous cavities due to changes of dimension in cooling and to the presence of absorbed gases. The dark areas are these cavities and cracks, the lighter areas are the ferrous sulphide, which contains the gold as the sharply defined round spots shown in the photograph. This separation

²⁷ Herbert Lang, *Matte Smelting*, p. 23.

²⁸ *Metallurgy of Silver*, p. 254 (1900).

²⁹ "Pyritic Smelting in the Black Hills," *Trans.*, xxx., 774.

³⁰ *Loc. cit.*, p. 39.

³¹ *Metallurgy of Silver*.

³² *Metallographist*, January, 1903, p. 26.

³³ *Trans.*, xv., 767.

³⁴ *Trans.*, xviii., 447.

³⁵ *Loc. cit.*, p. 768.

of gold agrees with the statement of Pearce referred to earlier in this discussion.

A portion of the ferrous sulphide was ground to an impalpable powder, passed through 100-mesh screen and vanned to separate as much gold as possible. The purified portion was then ground with mercury in a 2 per cent. solution of potassium cyanide to facilitate amalgamation, and finally washed by treating several times with 1 oz. of pure mercury. The residue was heated to drive off any residual mercury, and the percentage of gold estimated by assay. It still retained 1.044 per cent. of gold, which indicates that gold is not wholly insoluble in ferrous sulphide; but to prove this result conclusively would require a long and tedious operation to render it certain that no gold remained in an extremely finely divided condition.

The ferrous sulphide, together with all the gold, was fused with the addition of sufficient soft iron-wire to bring the resultant button up to Fe 68 per cent. Actually the button contained Fe 67.8 per cent. by analysis. This button containing an excess of iron showed no trace of a separation of gold, though it contained gold 6.288 per cent. by assay.

Two sections of this button are shown in Figs. 11 and 12, taken from the specimen after etching with alcoholic nitric acid of 2 per cent. strength. Three constituents appear to be present: 1, the metallic iron shows as black areas; 2, the ferrous sulphide shown as the light areas; and 3, a gray constituent which is shown under high power in Fig. 12 as the eutectic. In Fig. 12 the rounded areas are ferrous sulphide surrounded by the eutectic, and to one side is seen a body of iron as a bluish area. The constitution of this eutectic is not known. Its two components deport themselves with reagents as though they were iron and sulphide respectively, but, before etching, the dark component shows as a blue-gray, while the bodies of iron to which it seems allied appear as a bluish-white. A similar eutectic found in ferrous sulphide by Le Chatelier³⁶ and M. Ziegler is believed by them to be composed of oxide and sulphide of iron.

The gold is contained in the iron present and in the dark

³⁶ *Ibid.*, p. 28.

portions of the eutectic, as is proved by etching deeply with alcoholic nitric acid and then heating strongly when the golden color of gold replaces the dark areas of iron and the eutectic.

It is thus conclusively proved that though ferrous sulphide in the pure state does not exercise much solvent power upon gold, the presence of an excess of iron converts it into a substance of entirely different deportment towards gold. In this light it seems very probable that herein lies the explanation of the oft-times seeming erratic behavior of iron-mattes. The view which Carpenter takes of the functions of metallic iron in the furnace has been previously quoted, and certainly seems to be upheld by the facts.

Figure 13 shows a section of a typical iron-bottom. It was etched with 2 per cent. alcoholic nitric acid and shows four constituents: 1. Round or oval globules of matte included in the bottom as an emulsion. 2. Ferrite shown as the dark grains surrounded by a network. 3. A dark component of the network having an eutectic structure. 4. Bright, rod-like crystals of unknown nature, but resembling cementite. These crystals are not etched with nitric acid of 2 per cent. strength, and they are not coated with copper upon treatment with copper sulphate. The gold contained in this bottom is so small that efforts to detect its presence as compounds have been without success.

The question of the existence of double sulphides of copper and iron and also the mutual solubility of each in the other under varying conditions of temperature is now being investigated by a research.

It may be said that the evidence now available does not seem to favor the assumption of the combination of iron or copper sulphides to form double sulphides except perhaps, in very small quantity.

CONCLUSIONS.

At present the following provisional conclusions seem justifiable:

1. Mattes are not homogeneous, but are composed of distinct mineral entities, and may be likened to igneous rocks in which the mineral constituents fall out upon the lowering of the temperature and ensuing solidification.

2. The avidity of iron-mattes may be increased by increasing the reducing power and temperature of the furnace, thus obtaining a greater excess of iron in the matte produced.

3. The absorptive power for gold of a matte composed wholly of iron and sulphur is greatly dependent upon the excess of iron contained above that necessary to form ferrous sulphide (FeS).

NOTE BY THE SECRETARY.—Comments or criticisms upon all papers, whether private corrections of typographical or other errors or communications for publication as “Discussions,” or independent papers on the same or a related subject, are earnestly invited.

